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Vapour products — Determination of nicotine in vapour product emissions — Gas chromatographic method

Produits de vapotage — Détermination de la teneur en nicotine dans les émissions de produits de vapotage — Méthode par chromatographie en phase gazeuse

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Foreword

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This document was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*, Subcommittee SC 3, *Vape and vapour products,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 437, *Electronic cigarettes and e-liquids,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

In many countries, the regulation of vapour products requires reporting for nicotine compounds in emissions. Therefore, there is a necessity to have an International Standard in place to get reliable/comparable data on nicotine in electronic cigarette emissions.

This document was developed for the determination of nicotine in the aerosol from vapour products utilizing gas chromatography coupled with a flame ionization detector. The experimental design parameters[1][2] used to collect the aerosolised vapour should be evaluated and documented for each analysis.

The document is based on the CORESTA recommended method (CRM) 84[3], which was written on the basis of the results obtained in an interlaboratory study conducted in 2015 involving 18 laboratories[4] and an interlaboratory study conducted in 2019 involving 11 laboratories[5].

Vapour products — Determination of nicotine in vapour product emissions — Gas chromatographic method

1 Scope

This document specifies an analytical method to quantify nicotine of collected vapour product emissions by gas chromatography.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 20768, *Vapour products — Routine analytical vaping machine — Definitions and standard conditions*

ISO 24197:—,[1)] *Vapour products — Determination of e-liquid vaporised mass and aerosol collected mass*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
vapour product
device intended for human use, which normally contains electronic components that vaporize a liquid to generate an aerosol carried by the air drawn through the device by the user

[SOURCE: ISO 20768:2018, 3.1, modified – Note 1 to entry has been removed]

3.2
e-liquid
liquid or gel consumable which may or may not contain nicotine intended for transformation into an aerosol and then inhaled with an electronic nicotine delivery device

[SOURCE: ISO 20714:2019, 3.1]

3.3
aerosol collected mass
ACM
mass of aerosol collected on a glass fibre filter pad resulting from the operation of a vapour product by a routine analytical vaping machine after a defined number of puffs

Note 1 to entry: Routine analytical vaping machine is covered by ISO 20768.

1) Under preparation. Stage at the time of publication: ISO/DIS 24197:2022

3.4
puff block
finite series of sequential puffs as defined by the user or by the test request

EXAMPLE Puff block 1: puffs 1 to 50, puff block 2: puffs 51 to 100, puff block 3: puffs 101 to 150.

4 Principle

The vapour product emissions shall be generated and collected as described in ISO 20768. The aerosol collected mass is determined gravimetrically. The collected matter is then extracted with isopropanol solution containing internal standard(s). The nicotine content of an aliquot of the solution is determined by capillary gas chromatography with flame ionization detection (GC-FID), and quantitated by internal standard calibration. The nicotine content in the vapour product emissions is calculated. Results are expressed as the weight of nicotine per puff, per aerosol collect mass (ACM), or per puff block as warranted.

5 Reagents

Use only reagents of recognized analytical grade.

5.1 Carrier gas, helium (CAS 7440-59-7), nitrogen (CAS 7727-37-9), or hydrogen (CAS 1333-74-0) of high purity.

5.2 Auxiliary gases, air and hydrogen (CAS 1333-74-0) of high purity for the flame ionization detector.

5.3 Isopropanol (CAS 67-63-0), minimum purity 99 %, used with internal standard(s) to prepare the extraction solution.

5.4 Internal standards of high purity, quinaldine (CAS 91-63-4) or n-heptadecane (CAS 629-78-7) of purity not less than 99 %.

n-octadecane (CAS 593-45-3) or other appropriate internal standards may be used after assessment of their purity and determination that the internal standard does not co-elute with other components in the sample extract. The peak area of the internal standard on samples should be monitored for consistency. In cases where inconsistencies are found, analysis of a prepared sample solution without the internal standard should be performed to confirm the absence of a peak in the extract eluting at the same time as the internal standard.

5.5 Extraction solution, isopropanol (5.3) containing an appropriate concentration of the internal standard (5.4), this is normally in the range of 0,1 mg/ml to 1,0 mg/ml.

5.6 Reference substance, nicotine (CAS 54-11-5) of known purity not less than 98 %, or solution of nicotine certified reference material. Nicotine salicylate (CAS: 29790-52-1) of known purity not less than 98 % may also be used. Store the reference substance at a temperature in accordance with the manufacturer's recommendation.

The purity of the nicotine or nicotine salicylate may be verified in accordance with ISO 13276[8] or by any other validated method.

5.7 Calibration solutions.

Prepare a series of at least five calibration solutions with concentrations that cover the range of expected levels to be found in the test portion by adding weighed amounts of nicotine (5.6) to the extraction solution. The suggested concentration range is 0,05 mg/ml to 2,0 mg/ml.

Store these solutions between 2 °C to 8 °C and exclude light.

Solutions shall be allowed to equilibrate to ambient temperature before use.

Stability and storage time should be evaluated by the laboratory.

NOTE (22 ± 2) °C is generally found appropriate for ambient temperature.

6 Apparatus

Usual laboratory apparatus and, in particular, the following items.

6.1 Gas chromatograph, equipped with a flame ionization detector and a suitable data handling system.

6.2 Capillary column.

DB-ALC1[2)] capillary column (30 m length; 0,32 mm ID; 1,8 µm film thickness) has been found to be satisfactory.

Alternative capillary columns (e.g. a WAX[2] column or a DB-624[2] column) may be used provided that the peaks for solvent, internal standard(s), nicotine, propylene glycol, glycerol and other components in the sample extract are well resolved which can require optimization of the instrument conditions.

6.3 Vaping machine.

The aerosols shall be generated on a vaping machine in accordance with the specifications described in ISO 20768.

Standard conditions for puff duration and the puff profile are described in ISO 20768, other parameters may also be used.

7 Procedure

7.1 Preparation of test samples

All the vapour products shall be tested under the test atmosphere according to ISO 20768. Vapour products with rechargeable batteries should be fully charged before the test.

7.2 Glass fibre filter pads handling

Glass fibre filter pads shall be stored in the test atmosphere for a minimum of 24 h prior to determination of pre-testing weights.

For all operations, the operator shall prevent contamination from the fingers by wearing gloves of a proper material (powder free). Between operations, a glass fibre filter pad holder cap, if available, may be installed to prevent water loss or uptake. Glass fibre filter pads shall be processed as quickly after collection as is feasible to prevent uptake or loss of water.

7.3 Aerosol collection and sample preparation

The tested vapour product shall be operated in accordance with the manufacturer recommendations. Set up the aerosol trap system in accordance with ISO 20768, and collect the aerosol onto glass fibre filter pads in accordance with ISO 24197:—.

2) DB-ALC1, WAX and DB-624 are examples of suitable products commercially available. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

Aerosol collected mass (ACM) shall be determined gravimetrically in accordance with ISO 24197:—.

For each glass fibre filter pad, open the holder and remove glass fibre filter pad with forceps. Fold the pad twice with the aerosol side toward the inside of the folds by careful handling of only the edge of the glass fibre filter pad. Wipe the holder with the folded unused side of the glass fibre filter pad and transfer to a single sample vessel for extraction. Alternatively, wipe the inside of the filter holder front with two separate quarters of an unused conditioned filter pad and combine these in the same sample vessel.

7.4 Test portion

Extract each glass fibre filter pad using a suitable container and a fixed volume of the extraction solution (5.5) of 20 ml for a 44 mm pad, or 50 ml for a 92 mm pad, ensuring that the pad is fully covered. The volume of extraction solution (5.5) may be adjusted to give a concentration of nicotine appropriate for the calibration graph (see 7.7) provided that there is adequate volume for effective extraction of ACM. Analysis should be performed as soon as possible. The stability and storage time of the extracts shall be evaluated by the laboratory.

In instances where the samples exceed the upper limit of calibration, it is recommended to either dilute the samples with the extraction solution (5.5) or adjust the calibration range appropriately to quantitate the samples.

7.5 Setting up the apparatus

Set up the apparatus and operate the gas chromatograph (6.1) in accordance with the manufacturer's instructions. Ensure that the peaks for nicotine, internal standard(s) and other aerosol components are well resolved.

Suitable operating conditions are as follows.

- Oven temperature profile:
  - Initial temperature: 90 °C;
  - Initial hold time: 1 min;
  - Temperature ramp A: 15 °C/min;
  - Final temperature A: 120 °C;
  - Temperature ramp B: 40 °C/min;
  - Final temperature B: 280 °C;
  - Final hold time B: 2 min;
- Injection temperature: 250 °C;
- Detector temperature: 275 °C;
- Injection volume: 1 µl;
- Injection mode: split;
- Split ratio: 25:1;
- Carrier gas: helium, at a flow rate of 3 ml/min.

Optimize the gas chromatography conditions for analytes' separation and sensitivity. Once optimized, the same gas chromatography conditions shall be used for the analysis of all the standards and samples.